JOURNAL

OF THE

AMERICAN CHEMICAL SOCIETY.

VOL. X.—NO. 3.

MARCH,

1888.

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PUBLISHED MONTHLY.

NEW YORK:
JOHN POLHEMUS,
102 Nassau Street.
1888.

The Journal of the American Chemical Society appears monthly, except during July and August. It will contain all papers read before the Society, as well as abstracts from American and Foreign Journals, and a list of American Patents relating to Chemical Industries.

The Price of Subscription is $5.00 per year, in advance.

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A. A. BRENEMAN, 97 Water St., New York.

ON THE RELATIVE VALUE OF DIFFERENT PEPSIN TESTS.

BY JAMES H. STEBBINS, JR.

The gastric juice, secreted by the peptic glands of the stomach, contains two important principles, viz., hydrochloric acid and a ferment called pepsin.

By the combined action of these two principles it performs its physiological function, which consists in digesting albuminoid matters, *i. e.*, it disintegrates, dissolves and converts them into soluble, non-coagulable products, known as peptones.

Wasmann first isolated pepsin in 1839, but it must be said that his process, as well as those which have since been proposed for the preparation of this article, do not furnish perfectly pure pepsin. The product obtained is always more or less mixed with extractive matter, mineral matter and peptone, sometimes in considerable quantity, which our present knowledge of the art has not enabled us to completely remove. Perfectly pure pepsin is, therefore, not known, but we are nevertheless enabled to prepare products of very high digestive power.

Many methods have been proposed for testing pepsin, but none of them are perfectly satisfactory.

Thus, Bidder and Schmidt place a known weight of small cubes of coagulated white of egg in contact with a liquid containing a known weight of pepsin, dissolved in hydrochloric acid of 0.2 per cent. strength, and heat the mixture for about five hours at 45° C. At the end of this time the non-dissolved albumen is washed and weighed. The loss in weight of this albumen indicates the digestive power of the pepsin.

This method of assay has the inconvenience of taking into account only the amount of albumen dissolved, without paying attention to the amount of albumen really converted into peptone. The same applies also to the two following methods.

Grünhagen allows fibrin to swell in hydrochloric acid of 0.2 per cent. strength, and places the thick jelly thus obtained in a funnel,

closed at one end with a little glass wool, and exposes the whole in a drying oven to a heat of 45° C. After all excess of water has drained off, a certain number of drops of a solution, containing the pepsin under examination, is allowed to trickle upon the jelly contained in the funnel. In about two minutes the liquid will begin to drop from the funnel, and the rate of dropping in a given time is said to be proportional to the strength or activity of the pepsin.

P. Grützner determines the value of pepsins by allowing flocks of fibrin, stained with carmine, to digest under similar conditions, and then estimating colorimetrically, at the end of a certain time, the intensity of the coloration of the liquid separated by decantation from the non-dissolved fibrin, by comparing it with a certain number of standard solutions of carmine. It is very easy to stain fibrin evenly with carmine, and, therefore, the intensity of the coloration of the solution is proportional to the amount of fibrin dissolved by the pepsin.

The methods I propose to discuss in this paper are three, viz.: the U. S. P. test, the Manwaring test and the Kremel test.

According to the experiments of numerous investigators, the peptic digestion of albuminoids depends upon several conditions.

1. The temperature.

The pepsin of fish acts energetically at 20° C., but the pepsin of mammals requires a higher temperature, and it has been found that peptonization is most active between 35° C.–50° C. Above this, digestion runs much slower and ceases totally towards 70–80° C.

2. The quantity of pepsin.

There being no such thing as absolutely pure pepsin, it has been impossible to determine, with accuracy, the amount of albumen which can be converted into peptone by a given quantity of the ferment. We know only that the amount is very large, provided that from time to time a little acid and water is added in order to maintain a certain degree of dilution.

The quantity of albuminoid which can be digested in a given time increases rapidly with the quantity of pepsin employed till it reaches a maximum, and then decreases slowly. The quantity

of peptone finally obtained increases with the proportion of pepsin.

3. The quantity of water.

As the products of digestion accumulate, the rate of peptonization gradually decreases. The addition of a fresh quantity of acidulated water causes the peptic action to recommence until it has reached a certain limit, beyond which the reaction ceases entirely.

4. The nature and quantity of the acid used.

A large number of acids may take the place of hydrochloric acid in peptic digestions, but none of them are as efficient as the latter. A. Mayer found that with the use of hydrochloric acid, complete peptonization occurred in from 3 to 5 hours; with nitric acid in about 5 hours, with oxalic acid in 13 hours, and with sulphuric acid in 19 hours.

According to Brücke, peptonization is already very active in a medium containing only 0.8 pts. of hydrochloric acid per 1,000, and attains its maximum, with a concentration of 1 pt. of acid in 1,000 of water. A too large proportion of acid hinders peptonization, 7 pts. of acid per 1,000 of water being sufficient to make the action very slow. Mayer thinks that the most favorable proportion of acid is 2 pts. per 1,000 water, or 0.2 per cent.

5. The time of action.

6. The variety and character of the albumen.

One of the most largely used tests in this country is the U. S. P. test, which reads as follows:

"One pt. of saccharated pepsin dissolved in 500 pts. of water, acidulated with 7.5 pts. of hydrochloric acid, should digest at least 50 pts. of hard boiled egg albumen, in 5 or 6 hours, at 100–104° F. (37.5–40° C.)"

The above test seems simple, but, in reality, it is unreliable and misleading, as no two persons using the same pepsin can obtain the same or even approximate results; it is, therefore, not surprising that we meet with such a diversity of conclusions.

The weak points in the above test are the following:

1. The test is based upon the amount of albumen which can be dissolved in a given time (including peptone and intermediary products), but does not take into consideration the

amount of peptone actually formed, and this I claim to be of the greatest importance.

2. It directs that a given pepsin shall digest at least 50 pts. of coagulated albumen. Now, in order to determine how much albumen has actually been dissolved, it is necessary to use an excess of albumen, and then weigh what remains undissolved. The test in question does not specify how much albumen shall be used, but leaves it entirely to the option of the experimenter. I consider this to be a weak point, as it makes quite a difference whether only a small or large quantity of albumen is used.

3. It is difficult to see how accurate results are to be obtained by weighing the amount of undissolved albumen remaining after a digestion, because it is impossible to find two samples of coagulated albumen, which contain exactly the same quantity of moisture; and besides this, the quantity of moisture is very liable to vary during the weighing, owing to the loss of moisture by evaporation.

4. It is not stated how long the eggs shall be boiled! This is a very important matter, as digestion differs greatly according to whether the eggs are boiled for a short or a longer time.

5. No provision is made for the size of the pieces of coagulated albumen. This, also, is very important, as it has been found that the greater the surface of the albumen exposed to the peptic ferment, the greater will be the amount of albumen digested.

6. This test applies only to saccharated pepsins, and no provision is made for other brands of pepsin.

It will, therefore, be seen that the U. S. P. pepsin test is absolutely unreliable and misleading.

Lately my attention has been called to a new pepsin test, which I will designate by its author's name, the "Manwaring test." In this test Manwaring has tried to avoid as much as possible the bad points of the U. S. P. test; but in doing this he has stumbled against other sources of error which I will try to make clear further on.

The test can best be described in the words of its author:

"The design of the following mode of testing the dissolving power of pepsin is to conform as nearly as possible to the U. S. P. test, which, contemplating the testing of the saccharated form,

makes no provision for the proportion of acidulated water to be used with a pure pepsin.

"On the basis that 1 part of a pure pepsin is capable of dissolving 1,000 times its weight of coagulated egg albumen in 6 hours, a saccharated pepsin made with a pure pepsin of U. S. P. strength would contain 5 per cent. of pure pepsin; therefore if 1 grain of a U. S. P. *saccharated* pepsin is to be tested in the presence of 500 grains of acidulated water, then 1 grain of a pure pepsin should be tested in the presence of 10,000 grains acidulated water, to equal the same proportion of water and acid used for the *actual* quantity of pure pepsin contained in a U. S. P. saccharated pepsin when tested according to the U. S. P."

In order to render the weighing of small quantities of pure pepsin as easy as possible to the pharmacist, Manwaring recommends that it should be saccharated, and for this purpose he gives the following recipe:

R. Saccharated pepsin, consisting of:

Pure pepsin	1 grm.
Milk sugar	19 "

To make the test take of the above saccharated pepsin 0.3 grm. (= 0.015 grm. pure pepsin).

Coagulated egg albumen	22.5 grms.
Acidulated water consisting of:	
Distilled water 100 c.c. / Hydrochloric acid U. S. P. 1.25	154 c.c.

The eggs are to be boiled for 15 minutes and the whites pressed (by means of a spatula) through a (preferably flat) 30 mesh sieve. For the sake of uniformity, the egg whites should be cut into small pieces and thoroughly mixed before being passed through the sieve.

The mixture should be maintained at 100–105° F. for six hours, and agitated thoroughly about every half-hour.

At the end of six hours the temperature of the bath should be quickly run up above 145° F. to destroy the pepsin, then the bath with contained bottles allowed to remain undisturbed over night, that the undissolved albumen may settle.

If the test bottle has been kept securely corked during the test, or if by previously weighing bottle and contents and afterwards

making up with water any loss from evaporation, the quantity of albumen dissolved may be easily determined as follows :

From the settled contents of the test bottle pipette off 10 c.c. and evaporate to dryness—until weight is constant—in a watch glass. From this dry residue figure as follows (1 pt. of pepton or intermediate products representing 1 pt. of original albumen):

Suppose 10 c.c. of the liquid = 0.2 grm. dry residue ; $\frac{7}{1.4}$ times its weight = the quantity of water contained in the 10 c.c. that was derived from the albumen dissolved ; 10 c.c. of liquid less 1.4 c.c. of water leaves 8.6 c.c. water taken from the 154 c.c. of acidulated water in making the test.

1.6 grms. or 8 times 0.2 grm. dry residue = the quantity of albumen in its natural state as originally used, that has been dissolved in the 10 c.c. of liquid evaporated to dryness.

Therefore, if 8.6 c.c. acidulated water holds 1.6 grm. egg-albumen,
then 154 c.c. " " " 28.5 " "

Then, as 0.015 grm. pepsin dissolved 28.5 grms. coagulated egg-albumen, 1 pt. would dissolve 1900 times its weight.

The use of the multiplier 7 and 8 is based on the fact that egg-albumen averages 12½%, or $\frac{1}{8}$ dry.

As will be seen, this test is quite a departure from the U. S. P. test, and in some respects is an improvement upon the latter, but I object to it on several points, viz.:

1. It makes no provision for other than concentrated, or, as Manwaring calls them, *pure* pepsins, while in reality the number of these is small compared to the saccharated pepsins.

A person desiring to assay a saccharated pepsin by this test, would be at an utter loss to know how much pepsin should be weighed out, or how much acidulated water should be employed, unless he knew that the pepsin under examination was of U. S. P. strength.

This cannot be ascertained without submitting the sample to a complete chemical analysis, and hence a great deal of trouble and erroneous results are apt to ensue.

I have encountered this very trouble myself, as may be seen from the following figures :

A sample of pepsin, purchased as a pure pepsin (pepsin C in the tables), but which in reality was a saccharated pepsin, gave by this

method 420, *i. e.*, it is supposed to dissolve 420 times its weight of coagulated albumen.

Not knowing the strength of this saccharated article in pepsin, I was at a loss to know how much should be used in the test, and therefore decided to look upon it as a concentrated pepsin. In consequence of this decision on my part the pepsin was saccharated according to M.'s directions, and 0.3 grm. of this resaccharated pepsin used in the test, with the above result. The result obtained undoubtedly does the pepsin injustice, for it is probable that if I had not resaccharated the original article, and consequently diluted it to a much greater extent, that a much higher test would have been recarded.

The same applies also to the following pepsins, which were in the saccharated condition when bought and were resaccharated by me before testing.

Brand of Pepsin.	Grs. Albumen dissolved in six hours.
Pepsin A	550.6
" B	516.6
" C	420.
" D	398.6

2. We are further instructed to raise the temperature of the mixture after six hours' digestion up to and above 145° F. to destroy the peptic principle, and then the bath, with contained bottles, is allowed to rest over night. It has been shown by numerous experiments that the peptic principle is not killed at this temperature (145° F. = 62.2° C.), but that on the contrary digestion may and does continue up to 80° C., though of course much more slowly than at a lower temperature. If, therefore, a bottle containing a quantity of undissolved albumen be subjected to a temperature of 62.2° C., and that only for a comparatively short time (or during the time that the water bath is cooling), it is manifest that the peptic principle would not be injured, and that consequently digestion will progress all night. We should thus obtain a much too high and an erroneous result.

3. The next step in the process is to pipette off 10 c. c. from the settled contents of the test bottle, and evaporate to constant weight. Although I have found that as a rule the undigested albumen remaining in the test bottle settles pretty well over night,

yet there is always a little left suspended in the liquid, which, if pipetted off, as it must be, would add to the weight of the dry residue obtained from the 10 c. c. pipetted off, and hence would be a new source of error.

4. We are next directed to evaporate the 10 c. c. to dryness, and in doing this the residue chars or carbonizes, owing to the free hydrochloric acid contained in the water, so that in reality we weigh a certain amount of carbonaceous matter, instead of peptone, etc. Does this represent the amount of coagulated albumen dissolved by the pepsin? It may, but I strongly doubt it.

5. Assuming that the dry residue had not charred, what would we then be weighing? We would be weighing a mixture consisting of undigested albumen, partially digested albumen, and fully digested albumen, or peptone, together with all the intermediary products which always are to be met with in peptic digestions. Does this represent the efficiency of the pepsin, or is it not more likely that the amount of true peptone formed is an indication of the strength of the pepsin? The latter, in my opinion, is the more plausible explanation, especially if it be remembered that only the peptone is assimilable in the human system, while the intermediary products are not. In short, if we let the weight of all the products formed in a peptic digestion represent the efficiency of a pepsin, we would be obtaining a result considerably above what it ought to be.

6. As far as the calculations involved in this test are concerned, I would say that they are very ingenious and eminently fitted to make a pepsin test as high as possible. I will not dispute the accuracy of the multipliers 7 and 8, but do not believe that their accuracy is infallible in every test.

As this test was particularly gotten up to determine the digestive power of concentrated pepsins, I therefore append tests made by myself upon two such pepsins.

Pepsin E.—One grain of this pepsin was found to digest 819.2 grains coagulated egg albumen in six hours.

Pepsin F.—One grain of this article was found to digest 784 grains of coagulated egg albumen in six hours.

I have found by personal experience that the accumulation of peptone during peptic digestions hinders and finally stops the

action of the pepsin upon the albuminoid matter entirely. If now the liquid be diluted, digestion will recommence again and proceed until the pepsin has become inert. (I do not believe in the theory that one grain of pepsin can go on digesting to infinity.)

As Manwaring lays particular stress upon this question of dilution, I think his test is a decided improvement over the U. S. P. test.

The next good point in his test lies in the fact that he does not attempt to weigh the undigested albumen, as is done in the U. S. P. test, and thereby does away with a great source of error; but instead of this he figures the amount of albumen (?) digested upon a dry basis, and then tries to convert this dry basis by calculation into albumen on the wet basis. In doing this errors are apt to occur, as I have already pointed out, but I do not think that they are errors of such magnitude as are apt to be obtained with the U. S. P. test.

Finally, I wish to say a few words about a test which I consider to be the only approach to an accurate method of testing pepsin that I know of. I do not claim that this test is absolutely accurate either, as slight errors are apt to occur, which, however, do not materially injure the final result. I refer to the Kremel test, which was published some time since in the Druggists' Circular.

In devising this test Kremel has made a radical departure from the usual methods, and bases his test upon the fact that under the conditions in which artificial peptic digestions take place, pepsin alone has the property of converting albuminoid matter into peptone, and that, therefore, from an analytical as well as from a physiological standpoint, the only correct method is to take the quantity of peptone produced as a gauge of the action of the pepsin; or in other words, the test is made to resemble as nearly as possible the conditions existing in the natural process.

Without going into any further detail, the test is made as follows:

One grm. of egg albumen (soluble) dried at 40° C. and pulverized, and 0.1 grm. of the pepsin to be tested, are placed into a 100 c.c. flask, and dissolved in 50 c.c. of 0.2 per cent. hydrochloric acid. The solution is heated to 38–40° C. for three hours, and then exactly neutralized with sodium carbonate; it is then

heated on a water bath to 90° C., and cooled after coagulation has taken place. The flask is then filled to the mark with distilled water, and 50 c. c. are filtered off and evaporated to dryness in a platinum dish on a water bath.

The residue is dissolved in hot distilled water, filtered through a moist filter into a platinum dish, and the filter carefully washed. The solution is again evaporated to dryness and weighed. The peptone is then incinerated with ammonium carbonate, and the weight of the ash deducted leaves the weight of the pure peptone, or the representative of the digestive power of the pepsin.

The good qualities of the above test are the following :

1. Simplicity.

2. No guesswork, troublesome calculations or the use of questionable factors.

3. No weighing of albumen dissolved in hydrochloric acid, undigested albumen and intermediary products along with the peptone. This is all obviated by the use of soluble egg albumen, coagulation and filtration or removal of the undigested portion as detailed above.

4. The ease with which it is possible to duplicate and still obtain concordant results.

On the other hand, the objections to this process are the following :

1. The great difficulty of procuring absolutely pure soluble dried egg albumen. This source of error, however, in my opinion, is very slight, because in each test a large excess of albumen is always used, and consequently the pepsin always has enough albumen to act upon. Besides this it must be remembered that only the peptone formed is weighed, and not the amount of undigested albumen, as is the case with the U. S. P. test.

2. It may be objected to this test that the results obtained are expressed by the weight of peptone formed and not by the weight of albumen dissolved, and consequently the figures, being based upon dry peptone, will be much lower than when the result is expressed as so much moist or coagulated albumen. If this, however, be objected to, it is comparatively easy to obtain higher figures by a simple calculation. Assuming that the amount of dry peptone obtained is equivalent to so much dry albumen, then by

multiplying the weight of the latter by 8 (Manwaring's multiplier) we would obtain the equivalent in coagulated or moist albumen. I do not think it necessary or advisable to follow this course, as it involves the use of a multiplier which, as already pointed out, is questionable.

3. It takes a little longer to make a test by this process, but if accuracy is thereby gained the process is to be preferred.

To further illustrate the test, I append the following results obtained with commercial pepsins:

	Peptone formed from 0.1 grm. pepsin in 3 hours.
Pepsin G	0.5844
" E	0.4972
" B	0.4722
" F, crystal	0.4682
" C (saccharated)	0.4676
" H	0.4598
" A (saccharated?)	0.4370
" A (saccharated)	0.4246
" D plain, soluble	0.3470
" D pure, scales	0.3250
" D pure, another sample	0.3146
" I (saccharated)	0.2780
" J French	0.1848
" K (saccharated)	0.1738

These tests were all made with the same quantity of pepsin, whether the latter was saccharated or not, and I think are a fair indication of the relative values of the different pepsins.

It may be objected that this test does not do a concentrated pepsin full justice, on the ground that the latter would form a much greater proportion of peptone and thus retard if not completely arrest any further action of the pepsin upon the albuminoid matter.

In order to test this question, I saccharated samples of E, F and H respectively, according to Manwaring's directions, which is equivalent to diluting with mere acidulated water, and submitted them to the same conditions as before and obtained the following results:

	Peptone formed from 0.1 grm. pepsin in 3 hours.
Pepsin E	0.2620
" F	0.1240
" H	0.1250

It will be observed that in these tests the figures are considerably lower than in the former ones; but it must be remembered that the pepsins with which the tests were made were twenty times weaker, or rather more diluted, than in the previous tests, and notwithstanding this the peptone formed is proportionally larger than before. This would clearly show that dilution is beneficial in the case of concentrated pepsins, as it corrects the retarding action of peptone. As the dilution in these last tests was twenty times greater than in the previous ones, we ought, by multiplying each of the above results by twenty, to obtain the amount of peptone which would be formed by using the pepsins in their concentrated forms, viz.:

	Peptone that should be formed from 0.1 grm. concentrated pepsin in 3 hours.
Pepsin E	5.240
" F	2.480
" H	2.500

The above figures are not, however, obtained as has already been shown, and therefore the calculation is erroneous.

As all the results obtained by strictly following Kremel's directions are comparable among themselves, I do not see how the process can well be improved upon.

The mere fact that increased dilution increases the yield of peptone is not, in my opinion, sufficient reason for condemning the process. As the conditions prevailing in the stomach of a full grown man do not differ materially as to dilution from day to day, it is safe to say that pepsins of varying strength administered to such a person will only perform a certain amount of work and no more, and that, consequently, the results obtained by this test more closely resemble the conditions prevailing inside the stomach than any other.

In conclusion, it will be seen that all the tests mentioned in this paper are subject to faults and imperfections, some having more than others; and, therefore, all we can do under the present unsatisfactory state of affairs is to select the one which is least objectionable, and this, in my opinion, is the Kremel test.

A NEW CONDENSER ATTACHMENT.

By Joseph F. Geisler.

The condenser attachment consists of a bulb shaped cylinder about 2½ inches in diameter, and 4 inches in length (exclusive of lower contracted portion to fit into the stopper of the percolator, containing the extraction tube), into the neck of which a tube (1 inch in diameter), containing a syphon, is attached by fusion. The tube reaches well down into the cylinder, and is perforated above the height of the syphon, to admit the vapors into the condenser. The inner end of the syphon reaches very near to the bottom of the tube, and thus will thoroughly drain the same. The condenser attachment, made for me by Eimer & Amend, delivers about 15 c.c. of solvent at each syphoning, which is the most suitable quantity for ordinary work. Any desired quantity of solvent can be delivered by increasing the size of the reception tube, or raising the height of the syphon, in which case the size of the extraction tube must also be increased, to hold the increased quantity of solvent. The solvent drops from the condenser into the bulb shaped attachment and is discharged periodically by the automatic syphon upon the substance contained in the extraction tube below. The condenser attachment is of special advantage in the extraction of crude drugs, fats, alkaloids, resins, or substances to be removed by volatile solvents, such as ether, petroleum ether, chloroform, etc. One objection to the ordinary extraction tube and percolator with condenser is, that the time of extraction is unnecessarily prolonged by the continual dilution of the solvent in the extraction tube by the condensed portions from the condenser, before that already in the extraction tube has had time to drain off. The difficulty is obviated by the condenser attachment, for little of the condensed solvent will get into the extraction tube proper until the reception tube is filled to the bend in the syphon, when the tube empties itself. By a little care this can so be regulated that the solvent in the extraction tube is drained off before a fresh

supply is syphoned into the same from the tube above. By keeping sufficient solvent in the lower flask or receiver, there is no danger whatever of the material in the extraction tube becoming overheated and ejecting the solvent, when the latter is delivered upon the same. The inner tube of the Liebig condenser, used with this piece of apparatus, should have an internal diameter of $\frac{5}{16}$, or better, $\frac{3}{8}$ inch, and the lower end should be cut off obliquely. The following are the sizes of the different parts of the complete extraction apparatus, convenient and suitable for the needs of ordinary work:

1. A 6 oz. Erlenmeyer flask.
2. Extraction tube, $1\frac{1}{8}$ inches (inter. diameter) $\times$ 5 inches length.
3. Percolator, $1\frac{1}{2}$ inches internal diameter, $\times$ 7 inches for body of cylinder, exclusive of lower contracted end of about $2\frac{1}{2}$ inches. Total length of percolater, $9\frac{1}{2}$ inches.
4. Condenser attachment, size to deliver about 15 c.c. solvent.
5. Liebig condenser, about 15–16 inches in total length, with inner tube of $\frac{5}{16}$ to $\frac{3}{8}$ inch internal diameter, and lower end cut off obliquely.

ABSTRACTS.

ANALYTICAL CHEMISTRY.

Analysis of German Silver. FELIX OETTEL.

This alloy of Cu, Ni and Zn, with occasionally Sn, Pb, Fe, Co, and Mn, is most readily analyzed as follows:

Dissolve the alloy in nitric acid and evaporate the solution, previously filtering if tin be present, and adding 15 to 20 drops of sulphuric acid for each .5 grm. metal taken. Separate the lead as sulphate if present. The filtrate, free from Sn and Pb, is diluted to 100 c.c., 2 c.c. of conc. hydrochloric acid are added, and the solution is treated with H_2S gas. On completion of the precipitate, indicated by the rapid settling of the CuS, heat to boiling, cool and filter. Wash the precipitate with dilute hydrochloric acid containing H_2S, and

complete the washing with very dilute hydrosulphuric acid. Determine the copper as sulphide.

Evaporate the filtrate from the above to remove the HCl, take up with water, neutralize with KOH solution, add a few drops of sodium acetate (1:10), and then saturate with H_2S. Determine the precipitated zinc as ZnS.

Evaporate the filtrate to remove H_2S, oxidize with bromine water, precipitate the iron as a hydrate by NH_4OH, and from the filtrate, made strongly alkaline with NH_4OH, precipitate the Ni and Co by electrolysis.

Manganese, if present, is determined as Mn_3O_4. To determine the copper by electrolysis dissolve 5 to 6 grms. of the alloy in nitric acid, add 5 c.c. conc. nitric acid, dilute to 100 c.c. and submit to electric current.

Two samples thus assayed yielded :

Cu	61.22	57.53
Ni and Co	12.77	15.57
Zn	24.41	25.46
Pb	0.16	0.42
Sn	0.00	0.25
Fe	1.54	0.66
Mn	trace	0.14
	100.10	100.03

(*Zeit. anal. Chem.*, **27**, *15.*) J. F. G.

Generation of Hydrosulphuric Acid—Generation of Pure Hydrosulphuric Acid. CLEMENS WINKLER.

The author recommends barium sulphide for this purpose. 100 pts. of heavy spar, 25 pts. of coal, and 20 pts. of NaCl, all finely pulverized, are thoroughly mixed, and formed into a heavy paste by the gradual addition of water during the stirring. The mass is packed into a crucible, dried, and then ignited for a few hours at almost a white heat; cool, and finally remove the mass, and break into convenient sizes by the blows of a hammer. The BaS should be preserved in a dry place in well stoppered bottles or tin capsules. The H_2S is liberated from the BaS preferably by dilute hydrochloric acid. (*Zeit. anal. Chem.*, **27**, *26.*) J. F. G.

Detection of Sodium Phosphate in Glacial Phosphoric Acid. ANTON BETTENDORFF.

At 12° C. 1 part of sodium chloride requires for complete solution 1348 parts by wt. of hydrochloric acid of 1.190 sp. gr. At ordinary temperatures the transposition between the hydrochloric acid and phosphoric acid of the sodium phosphate is complete. Hence the presence of sodium phosphate in glacial phosphoric acid is indicated by the separation of crystals of sodium chloride when the glac. phosphoric acid is dissolved in hydrochloric acid of 1.190 sp. gr. (*Zeit. anal. Chem.*, **27**, *24.*) J. F. G.

Determination of Alumina in presence of Ferric Oxide and Phosphoric Acid. L. BLUM.

The author calls attention to the fact that dilute solutions of ammonium chloride are decomposed on boiling, the solution becoming acid in reaction due to liberation of the hydrochloric acid. To fully precipitate the aluminium as $Al_2(OH)_6$ by the addition of ammonium hydrate it is advisable not to boil the solution to rid it of the excess of ammonia, but to add a slight excess of ammonium hydrate, heat to boiling and filter immediately. (*Zeit. anal. Chem.*, **27**, *19.*) J. F. G.

Determination of Arsenic in Pyrites. H. FRESENIUS.

Comparative determinations of the arsenic in pyrites showed that the fusion method (fusion with KNO_3 and $NaCO_3$, etc.), offers no advantage in point of accuracy over the direct distillation of the arsenic with ferric chloride, after the preliminary oxidation in a stream of chlorine gas or with hydrochloric acid and potassium chlorate. (*Zeit. anal. Chem.*, **27**, *34.*) J. F. G.

Estimation of Chromium in Iron and Steel. J. C. ARNOLD and HENRY J. HARDY.

Precipitation as hydrate is open to the objection that phosphorus is usually present in the oxide finally weighed. Under suitable conditions chromium may be readily obtained as a basic phosphate of invariable composition ($Cr_6P_4O_{19}$) and may be weighed as such. The accuracy of the results depends upon rigid adherence to the details given. (*Chem. News*, **57**, *153.*) W. P. M.

Moisture Remaining in Gases dried by Means of P_2O_5. E. W. MORLEY.

Anhydrous phosphoric acid acts more thoroughly than concentrated sulphuric acid. It requires ten thousand litres of the dried gas to yield ¼ milligram of moisture, whereas this weight of moisture is contained in 100 litres of gas dessicated by means of sulphuric acid. (*Zeit. anal. Chem.*, **27**, *1.*) J. F. G.

A New Apparatus for the Direct Determination of Carbonic Acid. OSCAR OSTERSETZER.

This is a compact form of apparatus, in which the generated CO_2 is completely dried by passing through H_2SO_4, and further purified by passing through pumice stone saturated with $CuSO_4$, and finally absorbed in potassium hydrate solution and soda lime. With illustration. (*Zeit. anal. Chem.*, **27**, *27.*) J. F. G.

Gasometric Method of Determining Nitrous Acid. PERCY F. FRANKLAND.

The problem was to determine the nitrous acid present in a solution also containing nitrates, ammonia and organic matter. The quantity of nitrous acid present was too great to allow the use of any of the ordinary colorimetric methods, and the organic matter prevented the employment of standard permanganate.

Advantage was taken of the reaction between nitrous acid and urea:

$$2CO(NH_2)_2+2HNO_2=CO(NH_4O)_2+H_2O+CO_2+2N_2$$

and the calculation was based upon the volume of the evolved nitrogen. A tube, with cup and stopcock at one end similar to that of a Lunge's nitrometer was employed, and the introduction of the liquids was, as usual, made with aid of mercury.

An excess of crystallized urea was added to the residue left upon evaporation of the liquid under examination, 2 c.c. boiling water and the rinsings were added, and the solution was introduced into the tube. An excess of dilute H_2SO_4 (1 : 5), amounting to a few c.c., was then poured into the cup and admitted to the tube. An evolution of CO_2 and N followed. After 15 minutes, strong KHO solution was added and the mixture agitated until the CO_2 was absorbed. The volume of the remaining nitrogen was determined as usual.

In using this method it is always necessary, before beginning the evaporation of the original liquid, to add thereto a little strong KHO solution, as otherwise the salts of ammonium present would cause decomposition of the nitrites:

$$NH_4Cl+NaNO_2=N_2+NaCl+2H_2O.$$

Where nitrites and nitrates are each to be determined separately, the author proposes to determine the nitrites as above, and then, in a new portion, destroy the nitrites by evaporating with an excess of NH_4Cl and estimate the nitrate in the residue by the mercury method. (*Jour. Chem. Soc.*, **53**, *364.*) W. P. M.

J. Wiborgh's Gas-Volumetric Method for Determination of Carbon in Iron and Steel. H. V. JÜPTNER.

This method depends on the oxidation of the carbon by chromic and sulphuric acids, and the subsequent volumetric estimation of the resulting CO_2. For reasons discussed at length, treatment with cupric sulphate precedes oxidation by the mixed acids.

To obtain correct results, it is necessary that the carbon should be completely oxidized to CO_2, to which end it must be observed that (1) during treatment of the iron with cupric sulphate no carbon shall be lost as hydrocarbons; (2) that during oxidation with the mixed acids no hydrocarbons, but only CO_2, shall be allowed to form; and (3) that complete solution shall be effected.

[Detailed descriptions of method and apparatus are given.] (*Berg. u. Huttenwesen*, **36**, *67.*) D. W.

Tiles for Combustion Furnace. A. W. STOKES.

In place of the easily broken tiles of fire clay, the author greatly prefers slabs of asbestos cardboard. It is lighter, does not break, radiates less heat and cools more rapidly. (*Chem. News*, **57**, *150.*) W. P. M.

New Method for Determination of Ash. LUDWIG REESE.

Into a difficultly fusible glass tube A B, 40–50 cm. long and 18–22 mm. internal diam. and connected with an aspirator, is placed a smaller glass "combustion tube" *a b*, 16–20 cm. long and 11–15 mm. inter. diam., having one end contracted and plugged with a double thickness of platinum gauze. A coil of platinum wire

wound around the tube *a b* prevents the adhesion of the latter to the outer tube during combustion.

The material to be incinerated, contained in a porcelain vessel, is placed in the "combustion tube," a current of air drawn through the "combustion tube" by means of the aspirator, and by the proper regulation of the flames of a series of burners under the tube A B the drying and combustion proceeds very rapidly. (*Zeit. anal. Chem.*, **27**, *133.*) J. F. G.

Use of Asbestos in Filtration. W. FRESENIUS.

To facilitate the recovery and filtration of precipitates in liquid containing finely suspended matter, especially in digestion experiments, dilute the liquid considerably with water, add finely divided asbestos fibre, thoroughly mix by shaking, set the whole aside to settle, and, when clear, decant the supernatant liquid. Wash by decantation, and for recovery of the precipitated matter filter the residuum through a plug of asbestos fibre in a glass funnel. (*Zeit. anal. Chem.*, **27**, *32.*) J. F. G.

Apparatus for Fat Extraction. O. FOERSTER.

The improvement in this apparatus is in the form of the extraction tube into which the material to be exhausted is packed. The extraction tube has a cylindrical form, with lower end contracted, and the outer surface greatly thickened at a number of points, so as to allow a free circulation of the volatile solvent between the surfaces of the extraction tube and the percolator in which it rests, thus doing away with strings or wire necessary to suspend the tube in the more common forms of repercolation apparatus. (*Zeit. anal. Chem.*, **27**, *30.*) J. F. G.

Pressure Tubes. H. N. WARREN.

Glass combustion tubing (sealed at each end) is used as usual; the novelty being that the space between it and the outer (copper) tube is packed with magnesia. The whole is then immersed in a bath of melted lead. This arrangement is intended for temperatures ranging from 400° to 700° F.

For higher temperatures, as where a red heat is desired, the outer tube should be of wrought iron, and fine sand be substituted for the magnesia. (*Chem. News*, **57**, *155.*) W. P. M.

Pepper Adulteration and Analysis. F. M. Rimmington.

An illustrated paper of interest from a microscopic standpoint only. The author holds that no purely chemical process can be relied upon to detect modern pepper adulterations. (*Analyst*, **13**, *81.*) W. P. M.

Contribution to the Examination of Spices. Eugen Borgmann.

About 1 grm. of the pulverized spice is shaken with about 10 c.c. of water and the mixture poured upon an unglazed porcelain surface. The water is rapidly absorbed and leaves the spice in a separated condition suitable for examination by a lens. (*Zeit. anal. Chem.*, **27**, *31.*)

Examination of Cod Liver Oil and Vegetable Oils. Prof. E. Salkowski.

For determining the congealing and melting points fresh samples must be taken for each experiment. Of 11 cod liver oils tested all became turbid after 2½ to 3 hrs.; at —10 to —12°, all but 2 solid. Of the chilled oils placed in melting snow 6 became liquid (clear or slightly turbid) in 2 hrs., 5 remained solid. When the fresh oils, not previously chilled, were placed in snow 8 remained clear and liquid, 2 became turbid and thick and only one became solid.

By the Reichert-Meissel method 5 grms. of oil required from .1 to .2 c.c. of $\frac{n}{10}$ NaHO.

The peculiar color reactions with sulphuric acid (using the oil in chloroform solution) are due to the yellow coloring matter, cholesterin and the fatty acids of the oil. The first indigo blue developed by the action of the sulphuric acid is due to the yellow coloring matter which is characterized as a lipochrome. The yellow color can be extracted from the saponified oil by ether. Palm oil and the yolk of eggs contain it in large quantity, butter fat small amounts, cottonseed oil traces, and the other vegetable oils none.

The fatty acids of cod liver oil, freed from the yellow coloring matter, dissolved in 20 pts. of chloroform and treated with an equal volume of sulphuric acid, produce a deep brownish red mixture with a deep green reflection. To apply the test, decant the colorless chloroform after ½ hr. and pour a few drops of the sulphuric

acid in a few c.c. of glacial acetic acid. At first there is but little color developed, but on standing 1–2 hrs. a red violet color with a dirty green reflection results.

Cholesterin (melting point 146°) was found in each of the cod liver oils. Animal fats contain cholesterin, vegetable oils phytosterin.

The hot saturated alcoholic solution of cholesterin solidifies on cooling to a mass of crystalline plates which, under the microscope, appear as thin rhombic plates, frequently containing a re-entrant angle.

The phytosterin of vegetable oils, however, deposits from the hot alcoholic solution, in long needlelike crystals, arranged in star shaped bundles. If slowly crystallized the crystals may appear as elongated six-sided plates.

To separate cholesterin from cod liver oil take about 10 grms. of the oil and saponify with 10 grms. of KOH and 20 c.c. of dilute alcohol. After saponification dilute with water to about 600–700 c.c., shake with about 500 c.c. of ether, allow to separate, decant and evaporate the ether. The residue, if impure, is saponified with a little alcoholic potash and again extracted with ether. The ether solution is washed with water to remove any soap. Evaporate the ether, take up the residue with alcohol, concentrate to 1–2 c.c., allow to crystallize and determine the melting point of the purified crystals. A melting point of less than 146° would indicate the admixture of vegetable oil with the cod liver oil. (*Zeit. anal. Chem.*, **26**, *557.*) J. F. G.

INDUSTRIAL CHEMISTRY.

Free Carbonic Acid in Potable Water and its Effect on Leaden Pipes. E. REICHARDT.

Experiments show that it is only water containing free carbonic acid that attacks lead pipes. The view that lead pipes conducting such water become encrusted gradually, and thereby capable of resisting corrosion, has yet to be proved. Observation shows that, except with hard waters holding much lime, there is no deposit in the lead pipes, even after years of use. A water containing free CO_2 and proportionally rich in alkaline carbonates, in a pipe used 300 years, has caused a deposit of phosphatic lead oxychloride scarcely $\frac{1}{2}$ mm. thick, the pipe being in a good state of preserva-

tion. As to the occurrence of water containing free carbonic acid, different mountain springs usually do not contain more than that necessary for solution of the monocarbonate present, often scarcely sufficient to form bicarbonate, but sometimes more, especially in waters holding much lime in solution.

Experiments thus far show that these spring waters do not attack lead except to the extent of minutest traces.

As compared with the infrequent occurrence of free carbonic acid in spring waters, river waters much more frequently contain it, though in far smaller quantity. It has thus far been proven that waters containing bicarbonates either in very small or in considerable quantities, do not attack lead, and even free carbonic acid, in small quantities, is without effect in presence of much lime and magnesia. On the contrary, the less mineral content, or the "softer" a water is, the more readily is lead dissolved.

Distilled or carbonic acid free water dissolves lead slowly with separation of oxyhydrate; distilled water holding carbonic acid in solution dissolves lead in much larger quantity, with separation of basic lead carbonate, a separation which can be very complete.

Water, to be conducted through lead pipes, should, under all circumstances, be examined for free carbonic acid and the amount determined. Its action on lead plates should also be noted.

The method preferred for free carbonic acid is Pettenkofer's—by means of rosalic acid, with which a change of color is produced not interfered with by simple or bi-carbonates.

Pettenkofer recommends 1 part rosalic acid in 500 parts alcohol, adding baryta water until the solution acquires a red tinge. In proportion to the quantity of free carbonic acid present this solution loses its color or becomes yellow. If carbonates only are present the color becomes red to violet, and this is easily observable and determinable by neutralizing the free carbonic acid to the point where bicarbonate is formed.

It has been found that water containing free carbonic acid loses its solvent action on lead as soon as the formation of bicarbonate is effected; even with distilled water this takes place when the free carbonic acid has been neutralized by soda to bicarbonate.

1 cc. rosalic acid to 1 litre of water has been used in all experiments. (*Arehiv. de Pharm.*, **14**, *1049.*) D. W.

Abstracts of American Patents Relating to Chemistry.

(*From the Official Bulletin of the U. S. Patent Office.*)

February 21st, 1888.

378,095.—Apparatus for the manufacture of gas. A. C. Humphreys.

378,113.—Process of refining cotton seed oil. G. W. Scollay.

The oil is treated with a metallic chloride and an alkali.

378,114.—Process of refining cotton seed oil. G. W. Scollay.

The oil is treated with ochre and an alkali.

378,136.—Process of producing aluminum. J. S. Howard and F. M. Hill.

The ore is boiled with hydrochloric acid, and, after cooling, mixed with Spanish white or lime. The free acid is then evaporated off, and the mass heated to about 600° to volatilize the ferric chloride. The resulting product is charged in a lime lined crucible mixed with lime, charcoal, fluorspar, cryolite and sodium bisulphate, and the charge covered with stannous chloride and sodium chloride. The alloy produced is removed and melted in lead or bismuth, the aluminium and impurities skimmed off, and purified by heating on a bed of porous material.

378,230.—Process of treating liquids in evaporating pans. P. Casamajor.

378,231.—Process of treating cinchona bark. P. Casamajor.

The ground bark is separately treated with an acid and an alkali. The resulting solutions are mixed and the precipitate separated.

378,232.—Process of treating saccharine solutions. P. Casamajor.

378,233.—Treatment of cinchona bark. P. Casamajor.

The process consists in treating the barks successively with an acid, and with an alkali or alkaline carbonate, then precipitating the acids from the alkaline solution, and the alkaloids from the acid solution, and mixing the precipitates.

378,246.—Refining Canadian and similar petroleum oils. H. Frasch.

The oil is distilled with oxide of lead.

378,278.—Combining metals with aluminum. W. A. Baldwin.

378,326.—Apparatus for mingling and combining gases. A. C. Lewis.

February 28th, 1888.

378,490.—Process of manufacturing water gas. R. Boeklen.

378,583.—Insecticide. F. Fenstel.

An antiphylloxera consisting of tannic acid, ammonium sulphate, and potassium silicate.

378,673.—Apparatus for making sulphurous acid. C. E. Getchell.

378,681.—Apparatus for bleaching by electrolysis. E. Hermite and C. F. Cooper.

378,684.—Apparatus for manufacturing salt. H. A. Hogel and O. L. F. Browne.

378,774.—Process of treating sludge acid. H. de Grousilliers.

After floating off the petroleum and tarry impurities, sodium or potassium sulphate is added to the sludge acid. The resulting bisulphate is then dried and distilled.

March 6th, 1888.

378,940.—Apparatus for manufacturing gaseous fuel or illuminating gas. E. H. Shaw.

379,021.—Process of preparing glue stock. T. P. Milligan.

379,034.—Method of preparing and treating starch. J. C. Schuman.

379,090.—Process of refining oil. R. J. Wilson.

Intensely heated air is forced through the oil, and after the greater portion of the volatilized oil has been separated from the air, the air and uncondensed gases are utilized for illuminating or heating purposes.

379,150.—Dyeing animal textile fabrics with naphthazarin. R. Bohn.

The fabrics are treated with a chromium mordant and then dyed.

W. R.